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FURNACE DESIGN AND CALIBRATION OF A HIGH-TEMPERATURE CONTINUOUS PHOTOGRAPH POWDER X-RAY CAMERA

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FURNACE DESIGN AND CALIBRATION OF A HIGH-TEMPERATURE CONTINUOUS PHOTOGRAPH POWDER X-RAY CAMERA

by

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[redacted]

Submitted in partial fulfillment of the
requirements for the degree of

MASTER OF SCIENCE IN MATERIAL SCIENCE

from the

NAVAL POSTGRADUATE SCHOOL
June 1967

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ABSTRACT

Several modifications are described that were made to an x-ray camera for continuous powder photographs during programmed temperature changes. The original furnace was redesigned to extend the useful temperature range of the camera. Drawings delineating the size and operation of this furnace are included. All of the methods for furnace calibration are summarized, with special emphasis on the transition-melting point technique. Materials used for furnace calibration are tabulated for easy reference.

The camera with the new furnace was calibrated so that more precise high temperature measurements could be made. Further work disclosed that several more desirable features are needed; and these are given in the summary.

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1) Acknowledgements

My advisor, Professor N. W. Buerger, originally conceived the idea for this project, and continued to give advice and assistance for its successful completion.

Mr. Roy Edwards, Material Science and Chemistry Department Technician, provided many beneficial suggestions and services during the course of this research.

To Mr. Jerry Smith, Naval Postgraduate School Machine Shop, who manufactured the many parts needed for the furnace and helipot support.

My wife deserves special credit for the understanding and encouragement that was given during the difficult times of compiling and typing this thesis.

To Professor J. R. Clark, Professor G. D. Marshall, Mr. Bill Penpraze, and Mr. Don Clark, a sincere thanks for the assistance they offered during my association with the Naval Postgraduate School.

2) Introduction

Man's ability to conquer his environment and improve his living conditions has been determined in each age by the materials at his command. As better materials were needed, they were produced, and metallurgy kept abreast of technology. Unfortunately, in this Atomic-Space Age, we find that technology is far in advance of metallurgy in many respects. Already the designs of advanced missiles and supersonic aircraft are being limited by the non-availability of suitable materials.

One of the many tools available to man to narrow this technology-metallurgy gap is x-ray analysis. Since the discovery of the structures of the simplest crystals, rock salt or diamond, around 1913, the field has opened for a long succession of structure determination of more and more complex crystals. As the crystals investigated became more complex, greater effort was required on the part of the crystallographer to determine the structure accurately. To meet this challenge, x-ray analysis techniques were improved. Sir Lawrence Bragg recently defined x-ray analysis as the method of determining the arrangement of atoms in various forms of matter by observing how they diffract or scatter x-rays (4). This method is chiefly applied to crystalline materials, where atoms or groups of atoms are arranged in a repeating and regular manner.

In the early days of the development of x-ray analysis, the primary object was to determine the atomic arrangements in the metals and their alloys. The range of interest now extends far beyond this early goal in an attempt to keep up with technology. One of the problems engaging our attention at the present time is the behavior of metals and alloys at high temperature (5). High-temperature technology has demanded a rapid development of new high-temperature materials with specific physical and chemical properties. X-ray measurements at elevated temperatures offer the possibility of relating their high-temperature properties to structure. This can generally be accomplished from simply prepared powder specimens. Probably no single furnace design would be best for all aspects of the structure problem. Consequently, any new furnace should be designed for the specific x-ray measurement to be made.

One of the earlier cameras which gave good results up to about 600 degrees centigrade and at the same time was easy to manipulate was designed by N. W. Buerger, M. J. Buerger, and F. G. Chesley, and produced by Otto von der Hyde (6, 11). Cameras were subsequently developed which gave greater precision and higher temperatures, but they were basically of the same type. From all of these cameras, only one photograph at one temperature was obtained. This presented a tedious and time consuming task for the crystallographer if he were trying to determine a phase transformation temperature. To alleviate this problem, a radically new camera was conceived by Professor N. W. Buerger, and designed by Lieutenant Commander L. D. Keil (31). This camera would take a continuous series of exposures on a single sheet of photographic film, while the specimen was subjected to programmed temperature changes. This allows all of the x-ray diffraction patterns to be displayed on one film strip, making phase transformations readily apparent. The camera as designed and built has several limitations.

The objectives of this research are to develop and calibrate a high-temperature furnace that will allow more accurate phase transformation temperature determinations and more precise lattice parameter measurements.

3) Reference Review

A careful study of existing and proposed high-temperature x-ray furnaces was conducted before any attempt was made to redesign the existing furnace on the moving film camera. The requirements for a high-temperature camera that can be used for a variety of investigations are: furnace temperatures of 1000-1200 degrees centigrade must be attainable while maintaining a long heater life; temperatures of the sample must be accurately measurable; thermal gradients must be a minimum; furnace must be operable in any atmosphere; samples must be either solid or powdered; and the sample must be easily accessible (1). Obviously, not all of these features are required for each application.

Although furnace designs can be placed into four heating classes, external current heating is the only method adaptable to a wide variety of samples and is the only method considered for this camera (1, 6, 10, 11, 13, 16). External current heaters can be divided into two main classes--wire-wound and metal foil. Because of the limit imposed by the power supply, it was decided to use a wirewound external current heater.

The measurement of the temperature of the specimen has always constituted one of the principal problems in high-temperature x-ray work (21). Many ways of dealing with it have been devised, but criticisms may be made in respect to all of them. Perhaps the most widely used method is a measuring thermocouple (27, 28). There are many problems associated with their use and large errors can arise from the following factors:

1) differences in emissivity between specimen and thermocouple;
2) contamination of the thermocouple by material from the specimen;
3) conduction of heat along the thermocouple wire.

Of the three factors mentioned above, the cause of the largest error in the measured temperature is essentially loss of heat along the wire of the thermocouple. Many ways have been tried to reduce this error, which is considerable in the case of powder samples where the thermocouple cannot be in direct contact with the specimen (13).

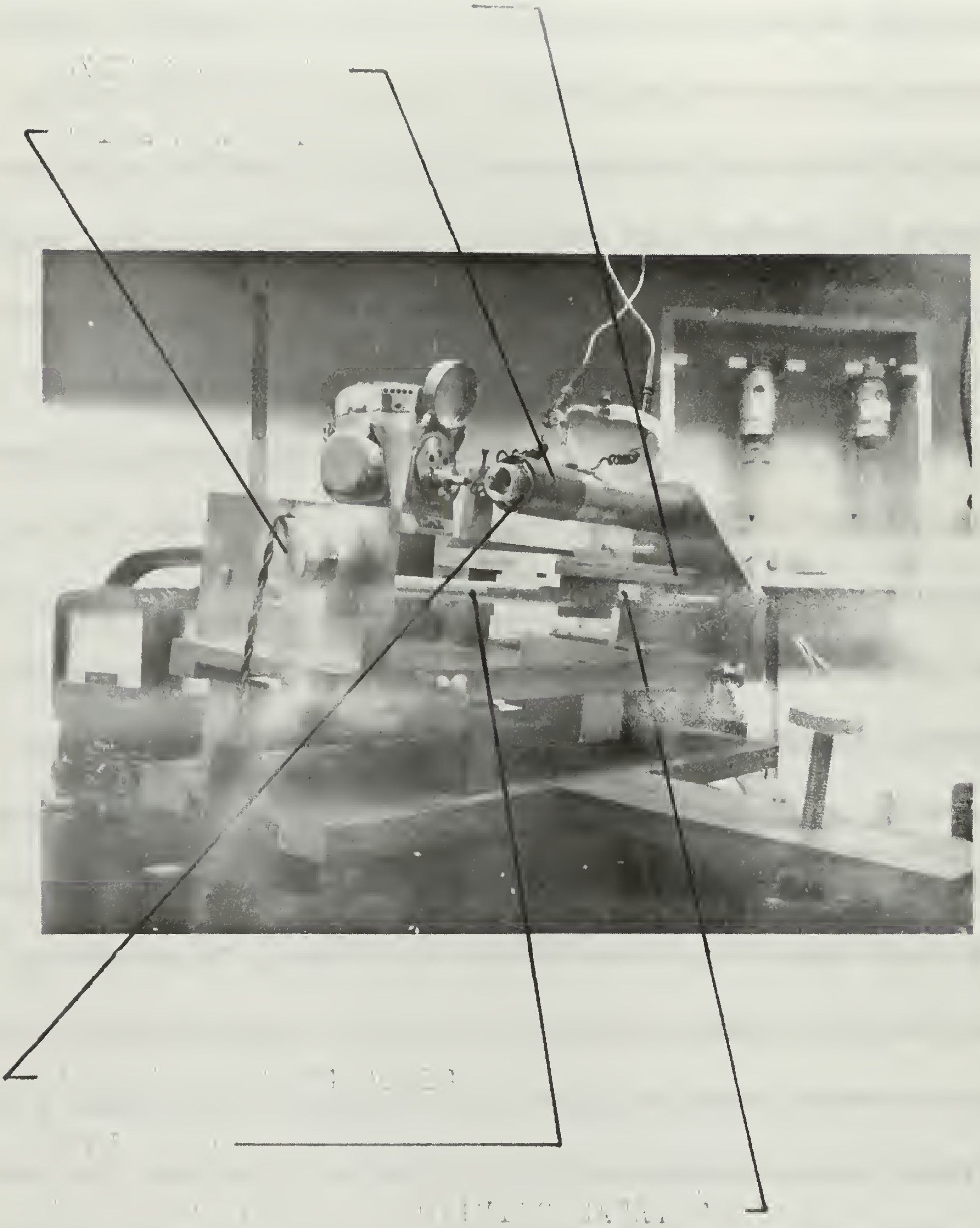
Other methods for arriving at sample temperatures for calibration of high-temperature cameras are:

1) calibration by power input
2) calibration by melting points
3) calibration by transition points
4) internal calibration
5) optical pyrometer

Calibration by power input is the simplest calibration method of those listed. The power input to the furnace is related to the temperature, derived from some other means, such as lattice spacing of a well known material. This is an excellent method for the calibration of a high resistance furnace, but can be quite unreliable when the room temperature resistance is approximately one ohm. Calibration by optical pyrometer is possible, but the accuracy is not high and certain obvious conditions must be fulfilled; the primary one being that the method works only in the incandescent range. Another method of temperature calibration is by the use of an internal standard. Some material, such as silver or aluminum, with well known expansion coefficients is mixed with the specimen before x-ray analysis is carried out. The major drawback to this method is that it cannot be used with other metals without alloying occurring. Considerable errors can also exist because the specimen may have different heat conductivity and emissivity than the internal standard (26). The calibration methods of melting and transition points are quite similar. One of the primary difficulties with these methods is that there are not many suitable materials with melting or transition points below 800 degrees centigrade. Although this method is more time-consuming than the other methods mentioned; the results are more certain, particularly in the case of the melting points. When the x-ray reflection disappears, that must be the melting point since the liquid cannot have the same crystal structure as the solid. A combination of several of these methods was used to calibrate the furnace and will be described in later sections.

4) Description and Operation of Apparatus

In order to describe fully the changes that were needed to make possible more precise measurements with the continuous powder photograph x-ray camera; it is necessary to review the camera as it was originally designed and operated (31). Figure 1 shows the camera assembly with the major components labeled. The drive rod for the moveable film carriage was driven by a small fractional speed synchronous timing motor manufactured by Bristol Motors of Saybrook, Massachusetts. This drive rod has 56 threads per inch which makes it possible to determine how far the film had advanced by simply dividing the threads per inch by the motor speed. For example, a 1/4 RPM motor would require three hours and 44 minutes to move the carriage one inch. The drive rod was designed with a fine thread so that the film would advance smoothly. No provisions were made in the event that the carriage momentarily stops due to slippage of the carriage on the drive rod or due to slippage between the timing motor and the drive rod. The timing motor itself doesn't run at exactly the rated speed. To prevent any errors from this source entering into temperature determination calculations; the device shown in Figure 2 was installed. It is simply a 5000 ohm linear helipot resistor with a gear attached to the pot. The gear fits into a rack soldered to the moveable carriage. Also mounted on the carriage is a standard metal ruler graduated in tenths of an inch. As the carriage advances, the helipot turns positively. A 20 volt D. C. power supply is connected to the helipot so that movement of the



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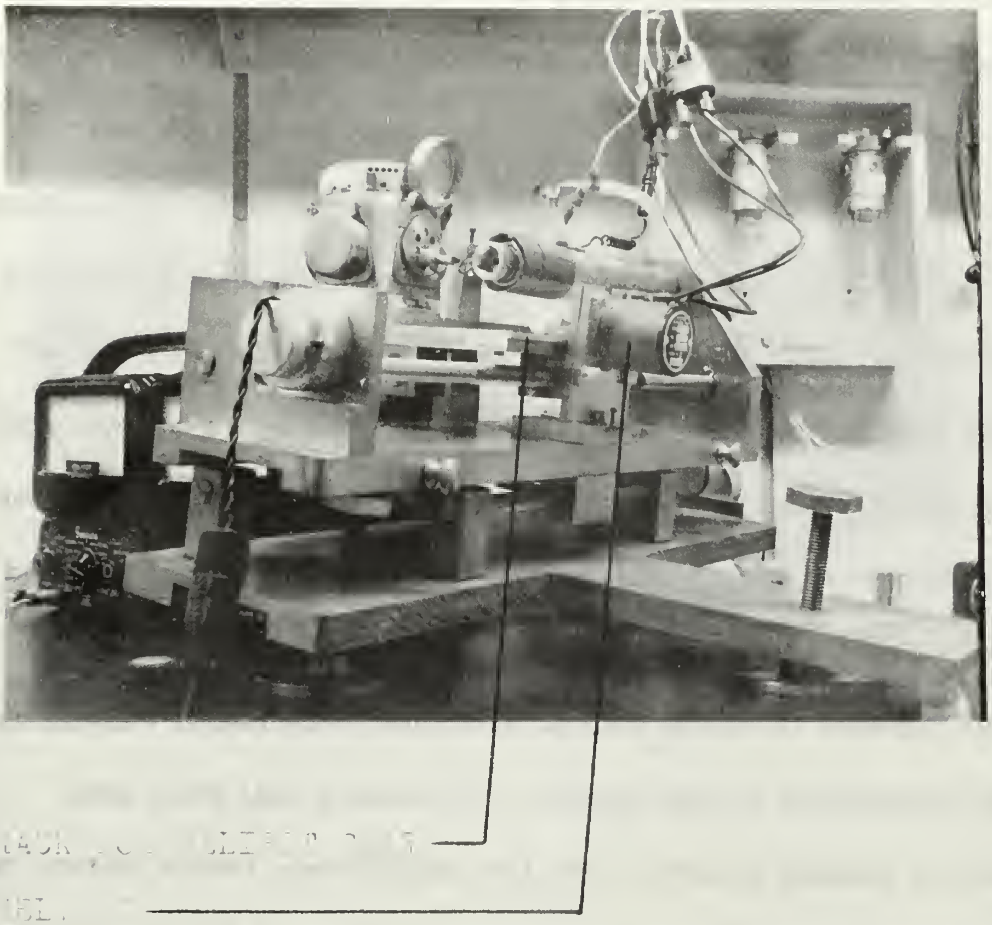


Figure . Helipot In Position for Film Travel Measurement carriage in inches may be recorded as voltage from the helipot on a suitable strip recorder.

An "O" ring and pulley arrangement on the shaft of another synchronous motor was used to operate the control knob of the furnace heater power supply. The rate of voltage increase with respect to control knob operation is described as being linear (29). Ten revolutions of the control knob produces the full twenty volt output, or two volts per revolution. Assuming that there is no slippage between the "O" ring and control knob, and that the rated motor speed is correct, this method gives accurate results. To remove the need for these assumptions, a set of Pickering 1:1 precision gears were mounted to the motor and to the control knob as shown in Figure 3. This assures that there is a positive increase of heater voltage with time. The heater voltage can also be recorded on a strip recorder. By using a dual track strip recorder of suitable accuracy, the film position and heater voltage can be correlated exactly for any position on the film. A strip recorder was not used for furnace calibration during this research project due to lack of a suitable available recorder. The lack of a recorder did not affect the precision of the results but did require more time from the investigator. The method used to correlate heater voltage with film position is shown in Figure 4. As each heated run was being carried out, voltmeters attached to the helipot and to the heater were monitored with respect to time. The starting position for each run was zero volts on the heater and 0.5 volts from the helipot output. As time increased the two voltages were recorded and plotted as in Figure 5. By knowing the

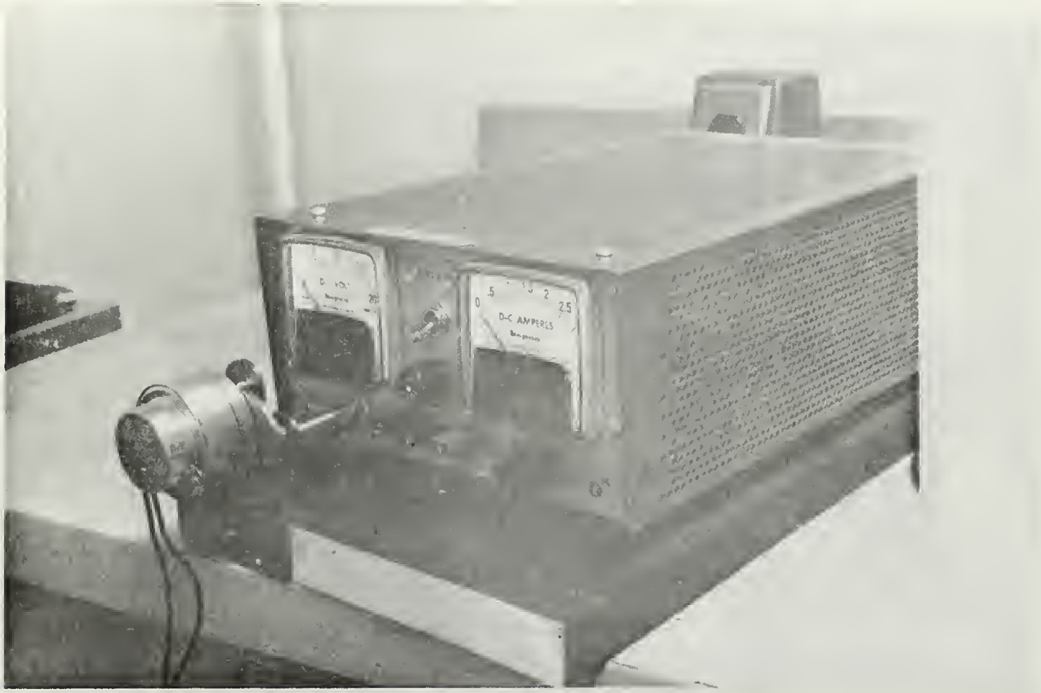


Figure 3. Heater Power Supply With Pickering Gears

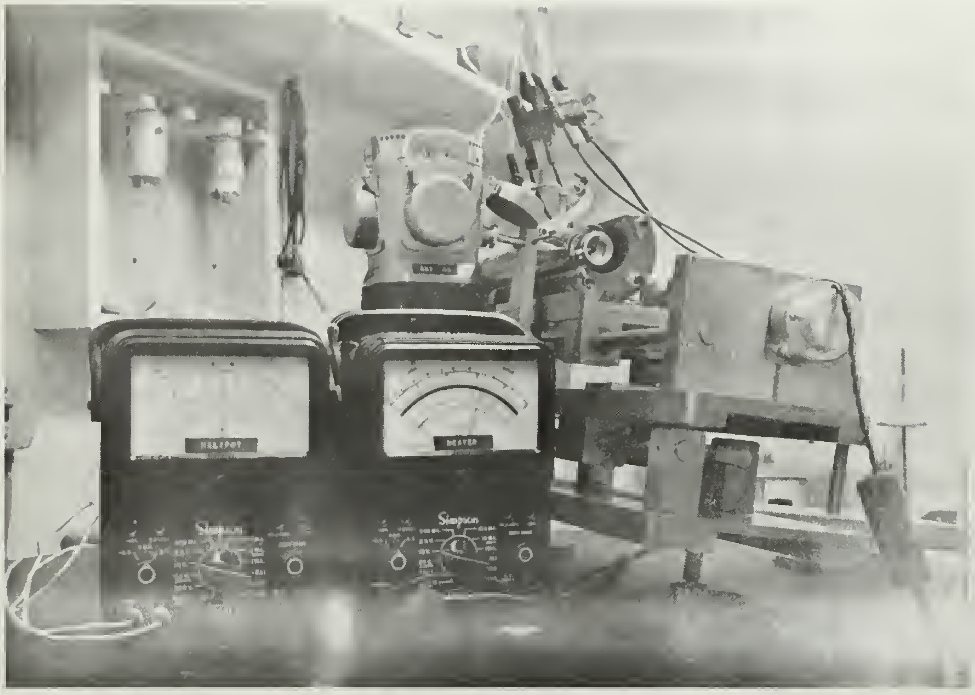


Figure 4. Voltmeters Connected TO Heaters And Helipot

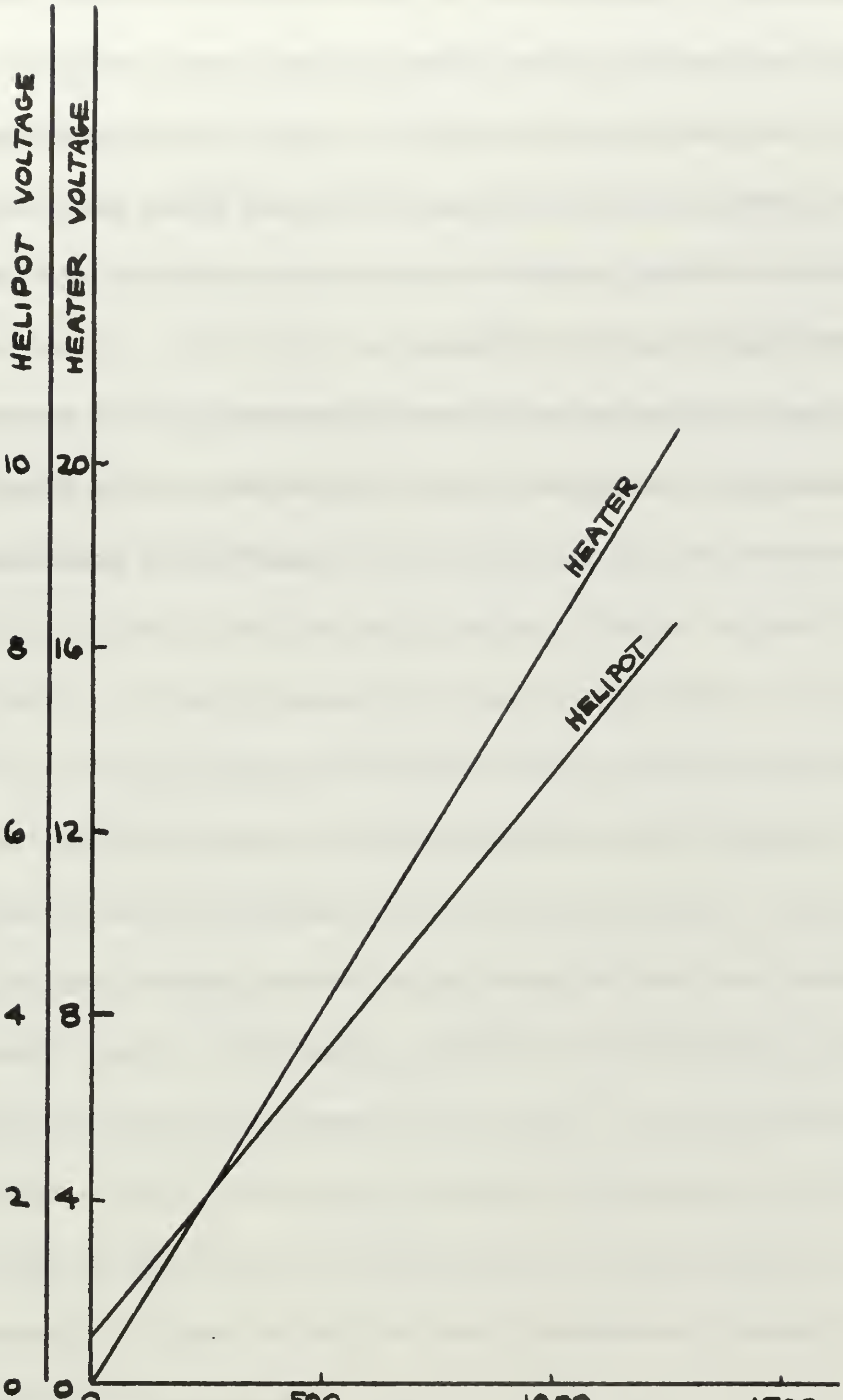


FIGURE 5. HEATER - HELIPOT CALIBRATION CURVE

helipot voltage at a particular point, it is possible to pick off the heater voltage quite accurately. The helipot voltage is determined by simply measuring the distance on the film to any point desired and multiplying this distance by the voltage increase rate. This was calibrated to be 1.40 volts per inch for the gear and rack arrangement. A check is possible to determine that the film and heater power supply are increasing smoothly by simply reading the two voltages and checking them against time on the plot of Figure 5.

Two other modifications were made to the camera. One was to the furnace which will be described in the next section; and the other to the film cassette which is discussed in the experimental procedures.

5) Furnace Design

The original furnace which was used with this continuous photograph powder camera was manufactured by Otto von der Hyde, and had been used for several years by the Material Science Department of the Naval Postgraduate School. It was a resistance type heater with a heating capacity of up to 800 degrees centigrade. The major components of this furnace were the water cooling jacket, heating element, radiation barriers, and mountings. In an effort to extend the usable temperature range of the furnace so that more materials could be examined, it was completely redesigned and remanufactured, with the exception of the water cooling jacket.

The goal which was set was to attain a furnace capable of heating specimens up to approximately 1000 degrees centigrade. This temperature was felt to be the maximum obtainable without using a vacuum system and still not have excessive oxidation of the heater element. With this goal in mind, the heater element was redesigned. One of the most stringent design criteria for the heater element was imposed by the power supply . The source of power for the furnace is a voltage/ current regulated direct current power supply. It was manufactured by Kepco, Inc., Mod ck 18-3 and is capable of delivering 20 volts and three amps at rated output. Because only 60 watts maximum is available for heating, the heater wire must have a resistance of exactly 6.67 ohms to reach peak temperatures.

Another design criteria was imposed by retaining the original water cooling jacket, which was necessary to prevent major redesign of the original camera. The water cooling jacket has a cavity into which the complete furnace is inserted for cooling. This cavity limits the size that the completed heater assembly can have, which in turn limits the heating element. The maximum size possible for the heating wire is a 9/32 inch helix one inch long. A final design criteria is that the wire selected must have a high melting point and a long life at operating temperatures.

To meet these design criteria, several wires of varying composition and size were evaluated for maximum temperature and life. The original goal of 1000 degrees centigrade specimen temperature requires that the melting point of the selected heater wire must be approximately 1400 degrees centigrade. Chromel A (melting point 1350 degrees centigrade) and Nichrome V (melting point 1400 degrees centigrade) were chosen as possible heater elements.

A 9/32 inch helix one inch long has a stretched length of approximately 14 inches, depending upon the closeness of the coils. To attain the required 6.67 ohms resistance, wires having a specific resistance of approximately 7±3 ohms per foot at room temperature were selected. The materials chosen for evaluation are tabulated in Table 1.

The resistance at elevated temperatures increases so that the room temperature resistance must be multiplied by a factor to determine the resistances. Rather than compute the needed lengths using this factor and then experimentally determining the correct length, it was decided to compute the room temperature length and then experimentally try for the perfect length. Using the above stated method, results

TABLE 1. SELECTED HEATER WIRES

WIRE TYPE	DIAMETER IN INCHES	B & S NUMBER	SPECIFIC RESISTANCE OHMS PER FOOT
NICHROME V	0.0113	29	5.02
NICHROME V	0.010	30	6.80
NICHROME V	0.008	32	10.0
CHROMEL A	0.0126	28	4.05
CHROMEL A	0.010	30	6.50

TABLE 2. WIRE LENGTHS REQUIRED

WIRE TYPE	ROOM TEMPERATURE COMPUTED LENGTH (INCHES)	MAXIMUM TEMPERATURE EXPERIMENTAL LENGTH (INCHES)
# 29 NICHROME V	15.95	15.75
# 30 NICHROME V	11.80	11.25
NICHROME V # 32	8.0	7.0
# 28 CHROMEL A	19.55	18.38
# 30 CHROMEL A	12.3	11.75

were obtained for many wires, and the average lengths are tabulated in Table 2.

Tests were run for each wire type at maximum temperature in an effort to determine their life. The only failures in a reasonable length of time were experienced by the number 32 Nichrome V. They tended to melt at any point where the air gap around the wire was small, so no further tests were run with this material. The other four wire sizes did not fail during test periods of as long as eight hours at full power. Because the camera is designed to use full power for periods not to exceed 1/2 hour, it was felt that this was a sufficient time length. Each of the wires was slowly heated from room temperature to maximum temperature many times with no failures encountered and no apparent change in maximum power used. In order to determine which of the wires to use in the first heater element, they were examined for oxidation. The number 29 Nichrome V and the number 28 Chromel A appeared to resist air oxidation at elevated temperatures better than the other types, so one heater element of each type was selected for initial manufacture.

The heater element consists of a helical coil of wire on the inside of a three piece, slotted, porcelain-brass tube which surrounds the specimen coaxially (Figure 6). The slot is necessary so that the x-ray beam may enter and exit without interference. The original design used a one piece porcelain tube with a circumferential slot of 290 degrees but it was felt that this made the element too fragile. In an effort to overcome this, three pieces were decided upon with the slot being cut

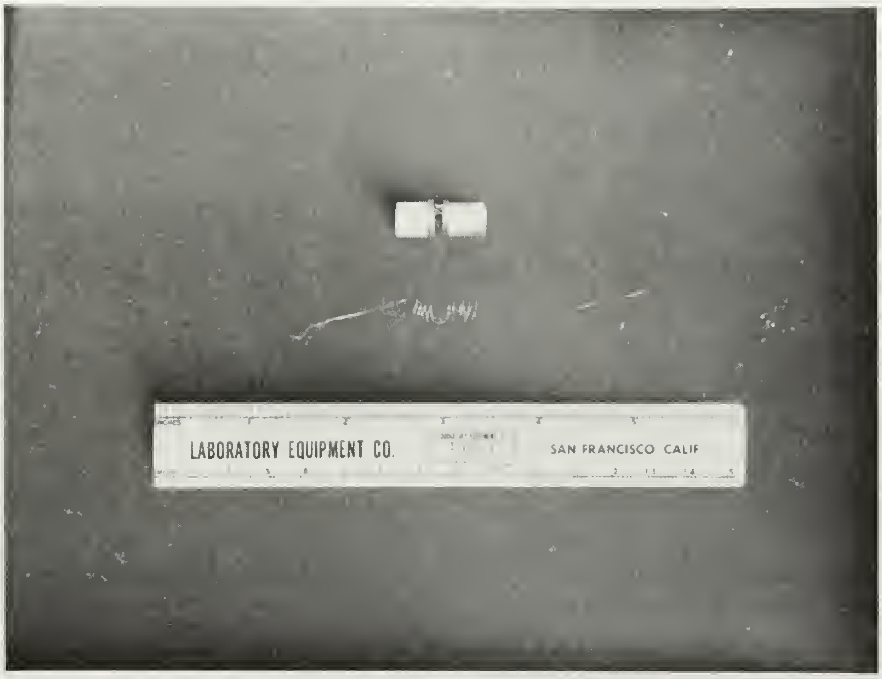


Figure 6. Heating Element

in a brass holder. This gave rigidity to the completed assemble as well as offering more protection from breakage. The helical coil of wire is in effect two helical coils with a reversal at the slot to allow the x-ray beam passage (Figure 6). To facilitate manufacture of these coils, the mandrel shown in Figure 7 was designed. It allows rapid forming of the coils with a removable screw in the center to affect the coil reversal needed. This mandrel offers a choice of two inside diameters for the coil. The smaller end forms a coil with an inside diameter of 0.220 inches while the larger produces 0.232 inches. Either inside diameter is satisfactory and the smaller was used for the number 29 Nichrome V, while the larger was used for the number 28 Chromel A because of the longer length of wire needed for the same resistance. Each helical coil is wound with a certain number of turns per side, and the experimentally determined figures are presented in Table 3 for each wire-inside diameter combination.

The porcelain-brass tube with its enclosed helical coil is inserted into two porcelain-brass endcaps, as shown in Figure 8. These endcaps were manufactured in two parts because of machine shop limitations, and could have been made in one piece from porcelain only. The endcaps also support the aluminum foil radiation barriers. Each brass part has two circular steps which provide air separation for the two aluminum foil cylinders. A heater unit was assembled and tested using the inner cylinder only; the outer cylinder only; and both cylinders; and a higher temperature was obtained using both cylinders (Figure 9). Each of these aluminum foil cylinders has two diametrically opposed holes so

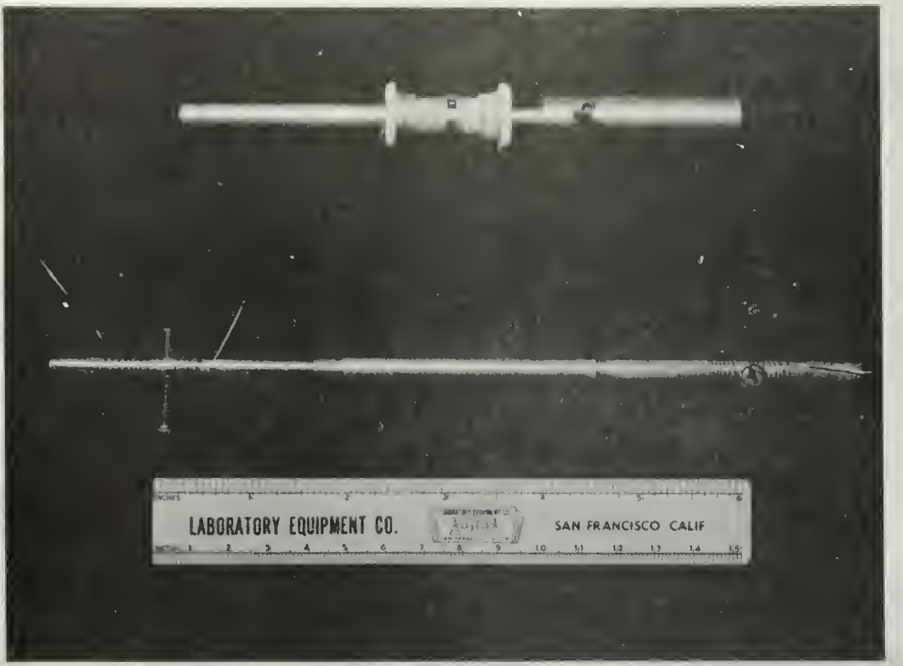


Figure 7. Helical Coil Mandrel And Radiation Barrier Jig

TABLE 3. HELICAL HEATER COIL DESIGN

WIRE TYPE	TURNS PER SIDE ON 0.220 DIA. MANDREL		TURNS PER SIDE ON 0.232 DIA. MANDREL	
	LEFT	RIGHT	LEFT	RIGHT
# 29 NICHROME V	8	8	7	8
# 30 NICHROME V	5	5	5	6
* 28 CHROMEL A	8	11	8	9
* 30 CHROMEL A	5	5	5	6

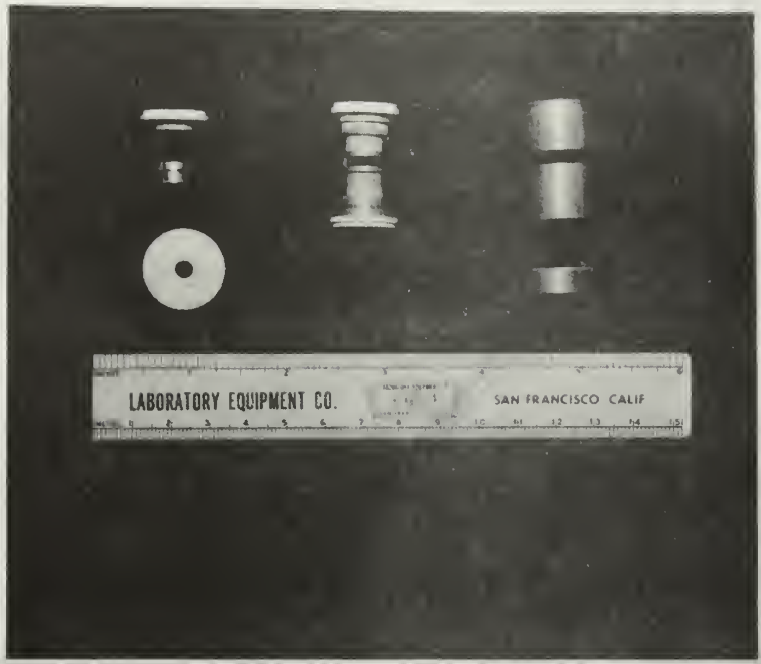


Figure 8. Heating Element And Brass-Porcelain End Caps

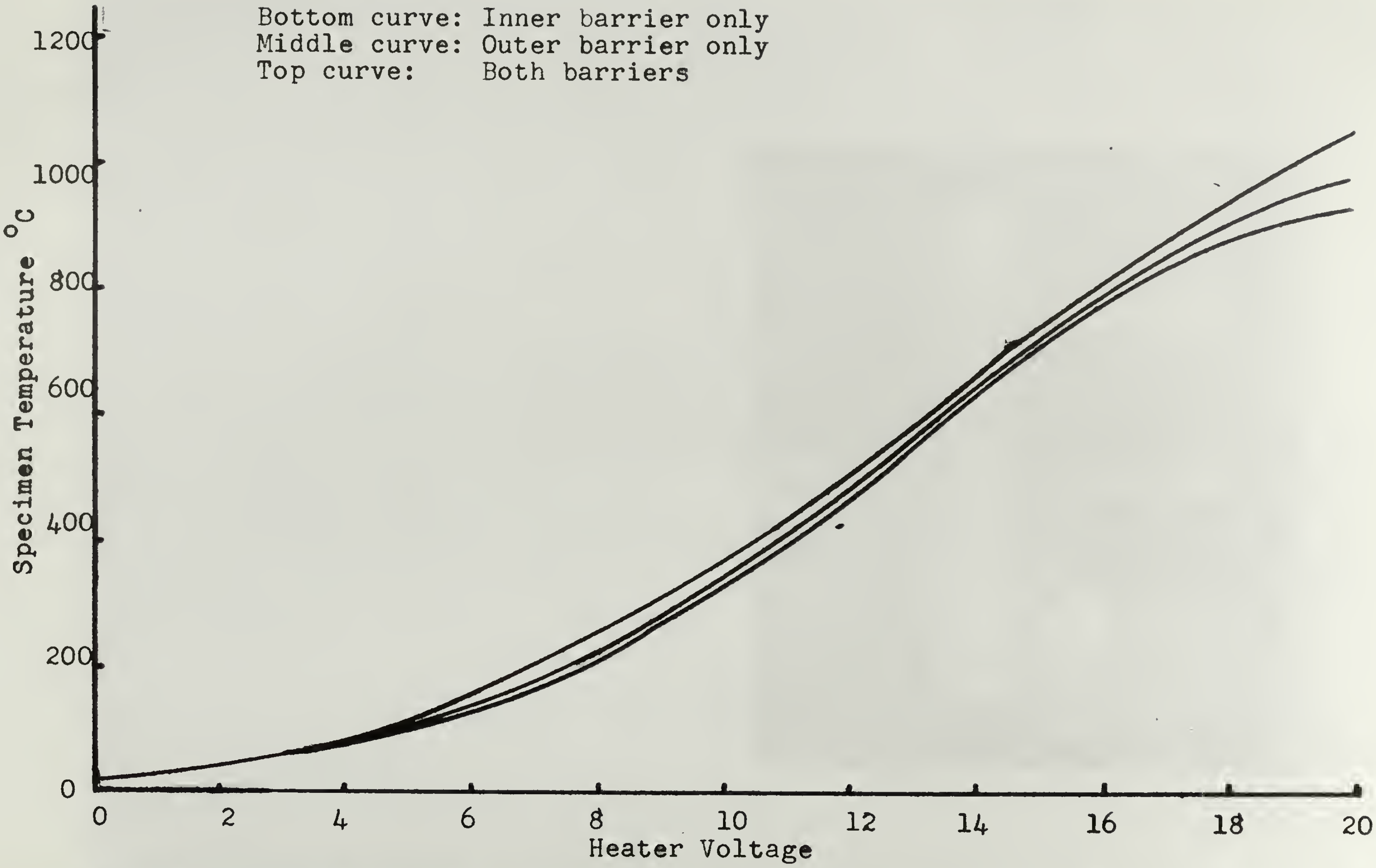
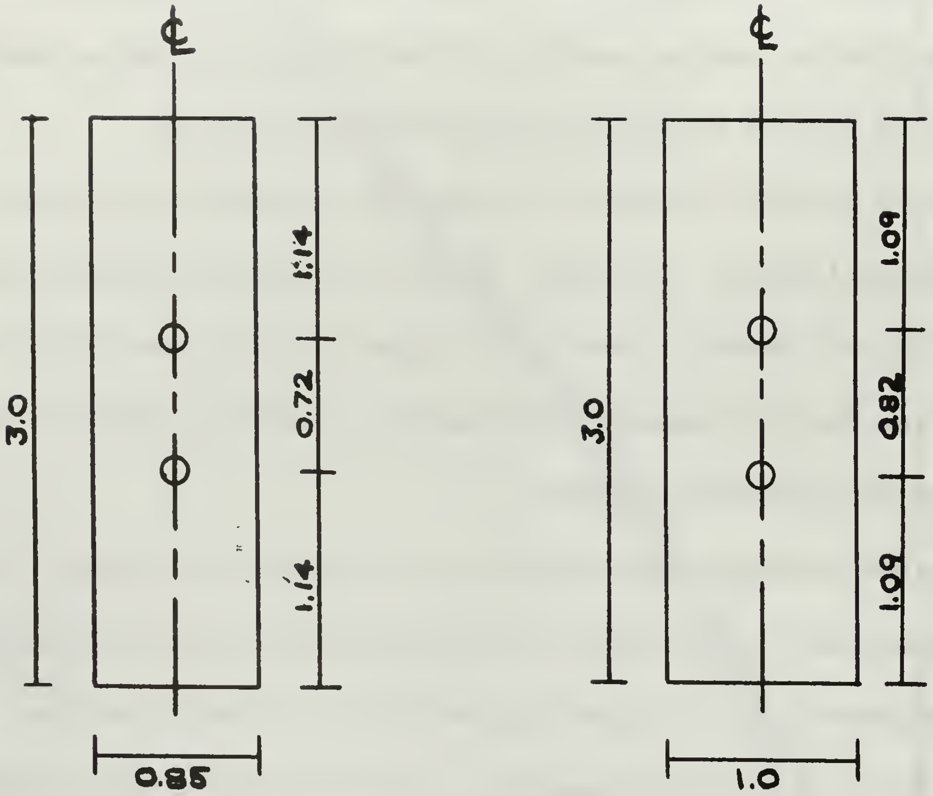


Figure 9. Chromel-Alumel thermocouple curves for various barrier combinations.

that the direct x-ray beam enters and exits cleanly. The inner foil cylinder was formed by cutting a strip of foil to a width of 0.85 inches and punching two 0.16 inch holes 0.72 inches apart on the longitudinal centerline (Figure 10). The outer foil strip was formed from a 1.0 inch strip with the holes spaced 0.82 inches apart. Both cylinders were separately folded onto the endcaps so that the folded material coincided with the 70 degrees of brass on the porcelain-brass tube.

The jig shown in Figure 7 was designed to facilitate the forming of the radiation barriers. The heater assembly is slipped onto the smaller end and is held firmly in place by the larger end by means of a set screw. The jig can then be clamped in any suitable position so that both hands are freed for the folding process.

The two cylinders were separately cemented using Techkits S-29 high temperature cement which was easily applied and dried quickly. This completed heating unit is then placed into a copper cartridge (Figure 8), for protection in handling. The slot in the copper cartridge must align itself with the heating unit slot, as well as the slot in the water cooling jacket in order that the diffracted radiation from the heated specimen may pass to the film. The copper cartridge has an open front and back so that the endcap contact is minimized; thereby reducing heat loss due to conduction. This heating unit cartridge is then inserted into the water-cooled jacket. One end of the heating coil winding is grounded to the jacket while the other end is insulated from the jacket by the porcelain endcap and a porcelain spacer (Figure 8). The



INNER BARRIER

OUTER BARRIER

Note: Allholes have 0.16 inch diameters.

Figure 10. THIN ALUMINUM FOIL RADIATION BARRIERS

completed unit is shown assembled in Figure 11 while the working drawings and parts list are given in the appendix.

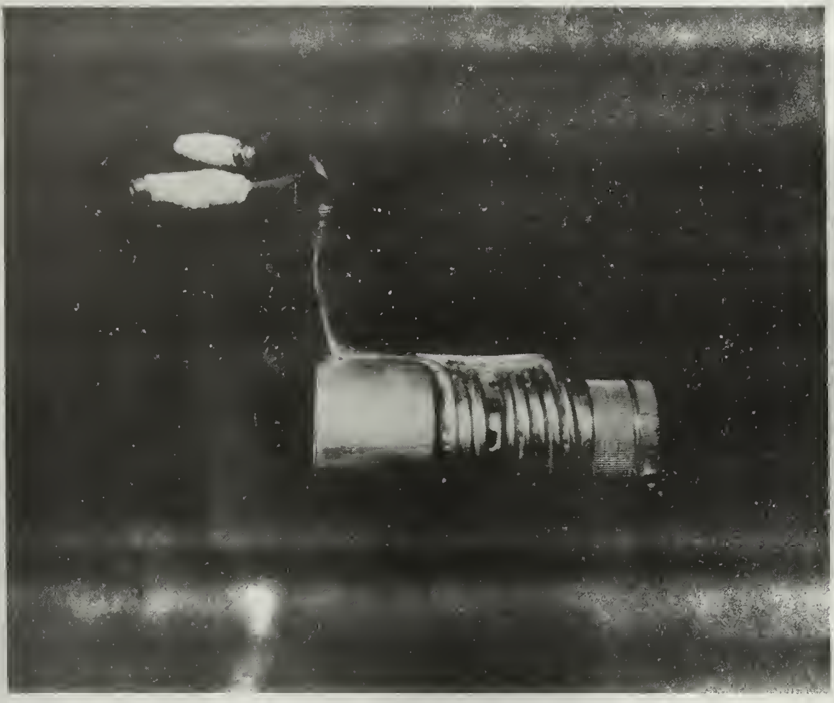


Figure 11. Combined Heater

6) Experimental Procedures

The furnaces that were designed and assembled had to be calibrated accurately so that temperatures could be determined with precision in relation to points of interest on the x-ray film. The test furnace had number 29 Nichrome V wire as the heating coil. As a first approximation to determine the possible maximum temperatures available from the completely assembled unit, a chromel-alumel thermocouple was used for temperature measurements. The results of several runs produced many scattered points, but the average curve shown in Figure 12 forecast a maximum of at least 900 degrees centigrade. Using this curve as a guide line, two types of furnace calibration materials were selected.

Metals: Pure metals with melting points in the range of 200-1000 degrees centigrade were selected as possible calibration materials (6). The metals that were available are given in Table 4. Powder specimens were prepared by using a clean jewelers file to produce small particles. These small particles were then screened through a 325 mesh bolting cloth to separate the larger pieces from the powder. The screened powder was mixed with a collodian or sealed in thin walled glass capillaries to become suitable specimens for room temperature powder photography.

The film cassette of the continuous photograph powder camera uses a 0.035 inch thick aluminum alloy sheet as a light shield for the film holder. Absorption of the reflected x-ray beam by the aluminum becomes a factor when the intensities of the reflected beam are low. To determine

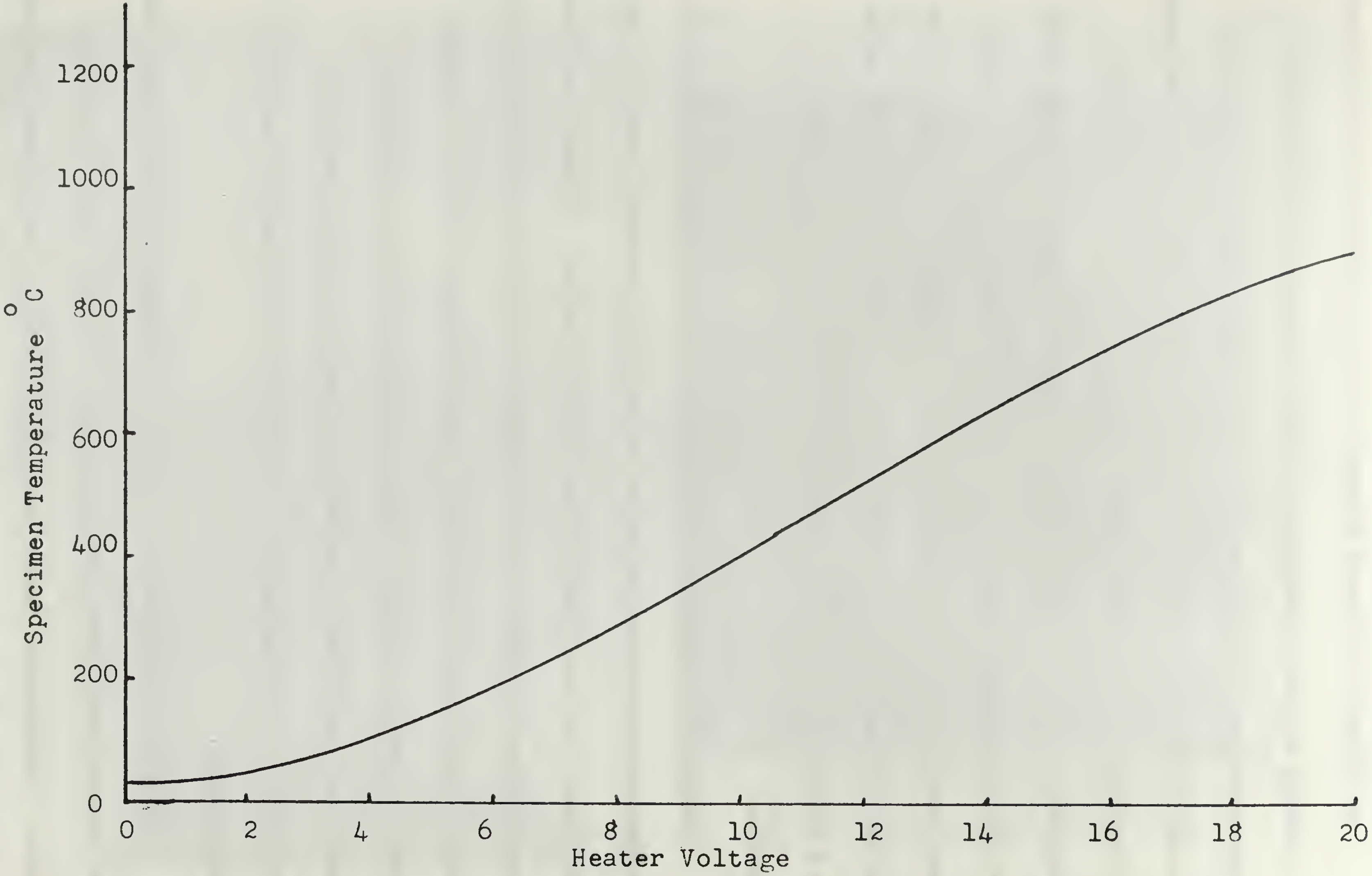


Figure 12. Chromel-Alumel Thermocouple Curve For #29 NICHROME V Furnace

Table 4. SELECTED METALS FOR FURNACE CALIBRATION

Pure Metal	Melting Point Degrees Centigrade
TIN	231.9 ± 0.1
BISMUTH	271.3 ± 0.1
CADMIUM	320.9 ± 0.1
ZINC	419.46
ANTIMONY	630.5 ± 0.1
ALUMINUM	660.2 ± 0.1
SILVER	960.5

which metals were compatible with this system, specimens were first photographed in a standard single exposure powder camera. In front of each film, a sheet of aluminum alloy was placed so that the line intensities produced would approximate closely those that should be seen in the moving film camera. In this manner it was determined that cadmium, bismuth, and antimony would be unsuitable as calibration materials. Specimen powder was prepared from the other three metals in much the same manner as previously described. Quartz capillaries distributed by Unimex-Caine Corp., Chicago, Illinois, were used to hold the powdered metal. The quartz capillaries were sealed using an oxygen-acetylene torch. The sealed capillaries prevented oxidation of the specimen and contained the metal when it melted. Figure 13 shows some of the prepared capillaries and their holders.

Non-Metals: In order to produce a smooth calibration curve for the furnace, non-metals with phase transformations or melting points below 1000 degrees centigrade were chosen (41). Table 5 shows some of the non-metals that could be used. From the long list of possible materials, many were not available. Potassium carbonate, ammonium chloride, sodium nitrate, and ammonium nitrate were secured for evaluation. These powders were ground up and screened through a 325 mesh bolting cloth before an attempt was made to prepare powder specimens. All of these powders were water absorbent and caked together after they were screened. Producing a uniformly packed specimen inside a quartz capillary was a slow procedure. The quartz capillaries used had an inside diameter of 1.0 mm. By using a capillary

Table 5. SELECTED NON-METALS FOR FURNACE CALIBRATION

Material	Structure Change	Melts
Potassium Carbonate	250 428 622	896
Ammonium Chloride	184.4	520
Sodium Nitrate	275	310
Ammonium Nitrate	32.1 84.2 125.2	169.6
Sodium Chloride		808
Sodium Perchlorate	308	
Barium Carbonate	810	
Sodium Carbonate	356 486 618	854
Quartz	573 867	
Zinc Sulfide	1020	

Note: All temperatures are in degrees Centigrade.

of 0.7 mm as a tamp, small portions of the powder at a time could be compacted into the 1.0 mm capillaries. The compacted powder was then sealed in the capillaries. The quartz capillaries are quite fragile and great care must be exercised when tamping the powder. As with metals, these specimens were placed in a standard powder camera with an aluminum alloy shield to determine which would be compatible with the moving film camera. Potassium carbonate was the only non-metal tested that produced weak lines, but it was decided to try it anyway, because it offered so many possible calibration points. Specimens that were prepared are shown in Figure 13.

Film Cassette Calibration: When an x-ray beam impinges upon a powder sample; it meets thousands of powder grains, each a tiny crystal in a different orientation. Among these grains many are so oriented that a particular set of planes makes the appropriate glancing angle θ with the x-ray beam. Each of these angles produces a curved arc on the photographic film. By measuring the distances between a pair of symmetrical arcs, the diffraction angles for a particular plane can be calculated using the following formula: $\theta = (\frac{180}{\pi} \cdot \frac{1}{4R})S$ degrees where S is the distance between symmetrical arc pairs and R is the radius of the camera. If the diameter of the camera is 57.3 mm or $\frac{180}{\pi}$ mm, then $\theta = S/4$ and we are able to measure the diffraction angle directly from the film with a millimeter scale. The continuous photograph powder camera was designed with a diameter of 57.3 mm.

Calibration of the cassette to correct the diameter to exactly 57.3 mm was done with 99.99 per cent pure aluminum. Powder specimens

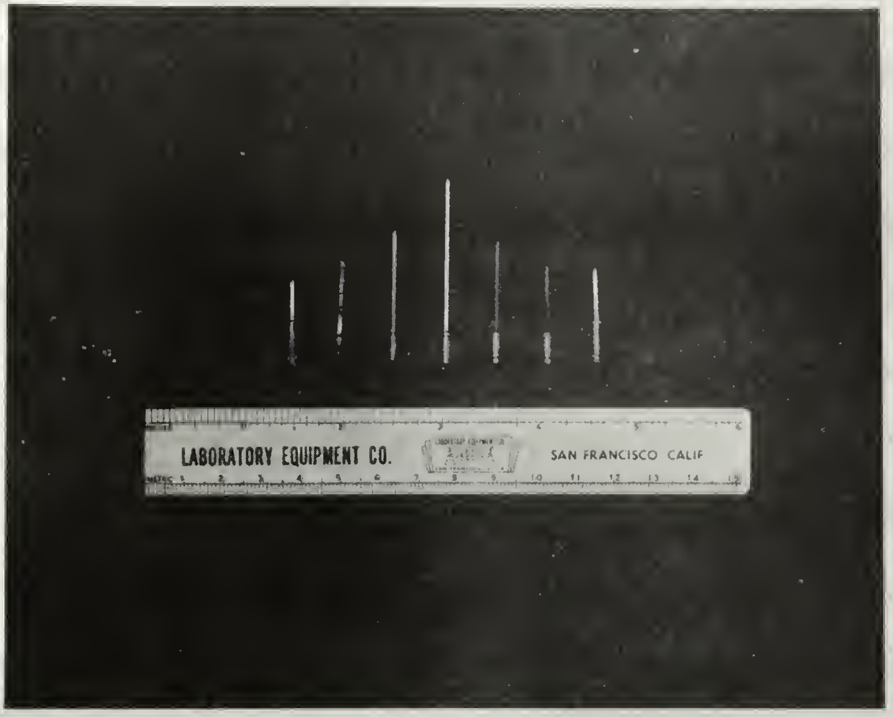
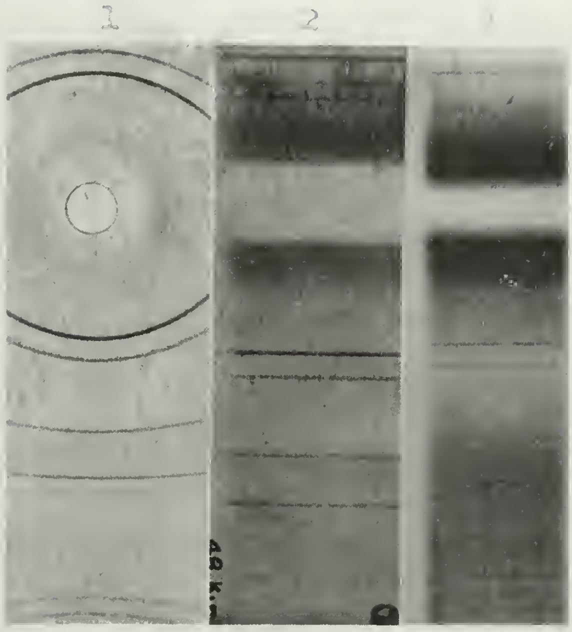


Figure 13. Powder Specimens In Capillary Holders

were made in the same manner as before and many x-ray photographs were taken with a standard powder camera at room temperature. The diffraction angles of all arcs visible were measured to within 0.5 mm and converted to degrees. The diffraction angles for the first seven most intense lines visible are given in Table 6. These angles were checked against those given in the Powder Diffraction File and were found to agree quite favorably. The same specimens were photographed using the moving film camera. The photographs from both cameras are shown in Figure 14. From these results it was determined that the cassette was not symmetrical about the powder specimen. As a preliminary step, the film carriage was raised 3 mm above the drive rod and another photograph taken. The effect of raising the front of the film carriage was to decrease the radius of the camera at $\theta = 0$ degrees and essentially keep the same radius at $\theta = 45$ degrees, which was shown to be needed by the first photograph. The radius of the cassette varied from 32.22 mm at $\theta = 0$ to 28.6 at $\theta = 55$ degrees.

Table 6. DIFFRACTION ANGLES FOR ALUMINUM

Plane	S(mm)	2θ(radians)
111	19.31	0.6740
200	22.46	0.7840
220	32.69	1.1411
311	39.20	1.3683
222	41.36	1.4437
331	55.83	1.9488
420	58.31	2.0354



1). Standard Powder Photograph
2). Uncalibrated Moving Film
3). Calibrated Moving Film

Figure 14. 99.99% Pure Aluminum Powder Patterns

7) Results

The results of the furnace calibration can best be shown photographically. Not only are the points used for calibration shown, but the versatility of the camera itself is demonstrated. Figure 15 shows the phase transformations of ammonium nitrate. Each of the three transformations are clearly visible. The transformation temperatures are listed in Table 5. It was experimentally determined that it took 1.15, 3.5, and 4.4 heater volts respectively to cause the three transformations.

Figure 16 shows the transformation point of ammonium chloride. A heater voltage of 5.17 volts was required for this phase change. The melting point of sodium nitrate is shown in Figure 17. The voltage required for melting was 7.3 volts. A phase transformation is shown as a gradual slope rather than an abrupt point. This gradual change might be missed on a standard single photograph high temperature camera, but is readily apparent on a continuous photograph.

The melting point of zinc is clearly visible in Figure 18. A temperature of 419.6 degrees centigrade is produced by 9.55 heater volts. Figure 19 shows the melting point of aluminum. The heater voltage of 13.5 volts for aluminum was determined, as were all the other above transition-melting points, by the method described under Operation of Apparatus. Many materials were tested in an attempt to find a transition or melting point in the vicinity of 900 degrees centigrade. This was necessary to prove that the heater was linear at higher voltages. The following materials were tested with no significant results: quartz, pure iron, and barium chloride. Pure silver finally

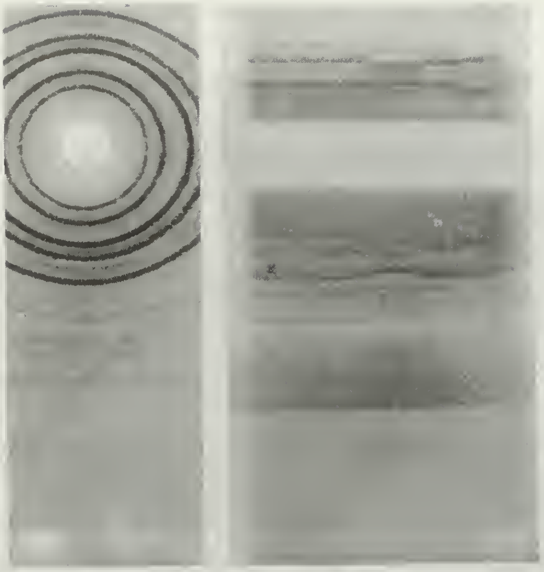


Figure 15. Ammonium Nitrate Powder Patterns

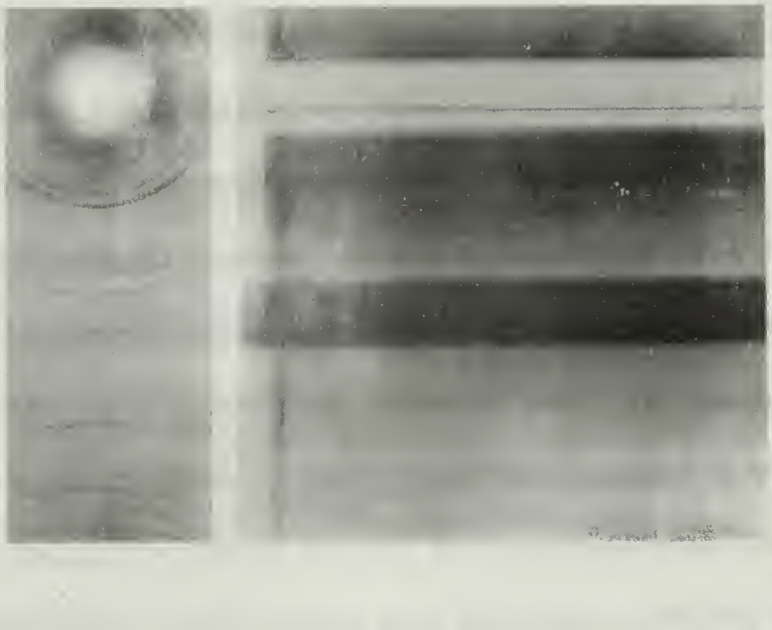


Figure 16. Ammonium Ch [illegible]

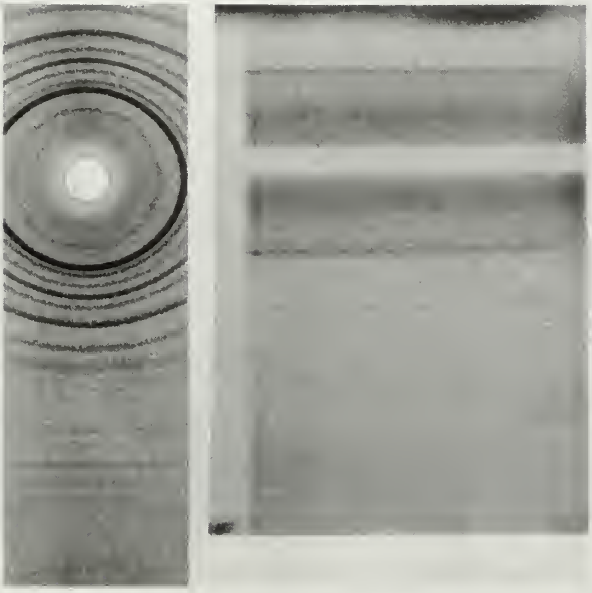


Figure 17. Sodium Nitrate Powder Patterns

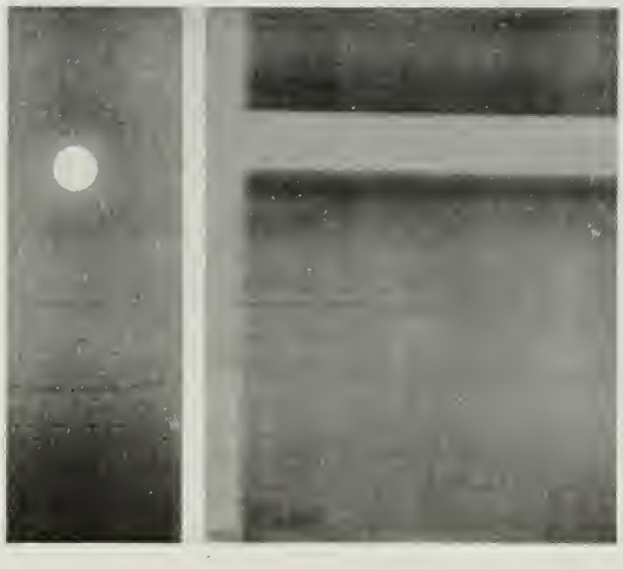


Figure 18. Zinc Powder Patterns

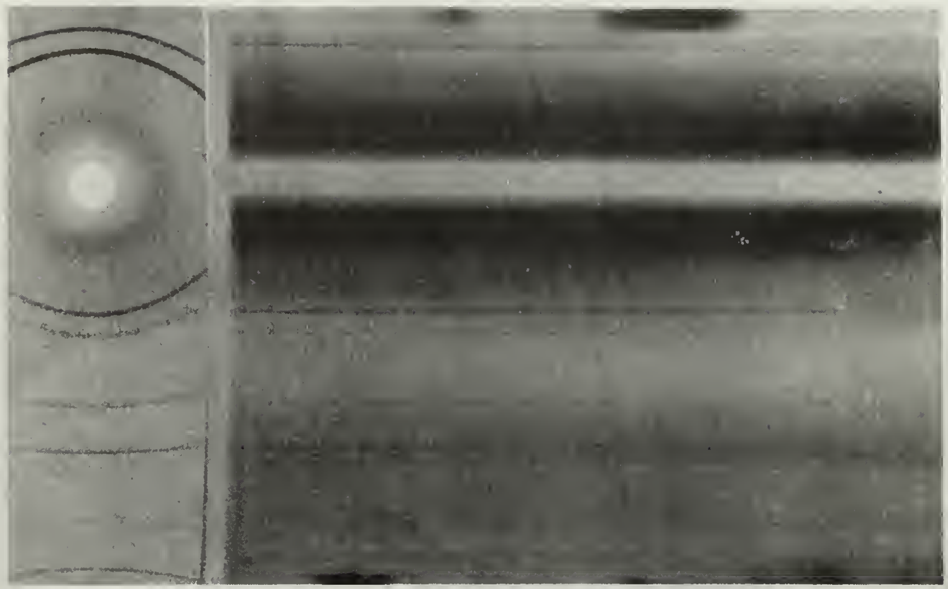


Figure 19. Aluminum Powder Patterns

gave a melting point at 18.3 heater volts; and zinc sulfide gave a transition point at 19.4 volts. This allowed a smooth calibration curve to be drawn.

Figure 20 shows the complete calibration curve. The furnace was found to be non-linear up to a temperature of 185 degrees centigrade, but then increases linearly at the rate of 62.00 degrees for each volt increase in the heater power supply. At higher temperatures, the curve is still linear, instead of tailing off as the thermocouple curve did. Because the rate of voltage increase is linear with temperature increase, it is possible to determine transformation temperatures within ± 5 degrees centigrade. The maximum temperature that can be produced is 1045 degrees centigrade which is produced by 19.75 heater volts.

The film cassette was calibrated as closely as possible without actually redesigning the film carriage and drive rod block. The film carriage was raised and lowered in very small increments with an Al powder photograph being taken at each step. Calculations were made from each photograph using the relationship $4\theta = S/R$ radians. Table 6 gives θ in radians for the Al lines used. It was found that when the carriage had been raised 0.12 inches that the diameter of the camera was 57.23± 0.1 mm as compared to a desired value of 57.26 mm.

Precise measurement of lattice constants is difficult due to the lack of a back reflection pattern. Systematic errors decrease with increasing θ and become error-free at $\theta = 90$ degrees. The maximum θ value possible due to cassette geometry is 60 degrees. Therefore, it is felt that the diameter of 57.23± 0.1 mm is not accurate enough for cell edge determination.

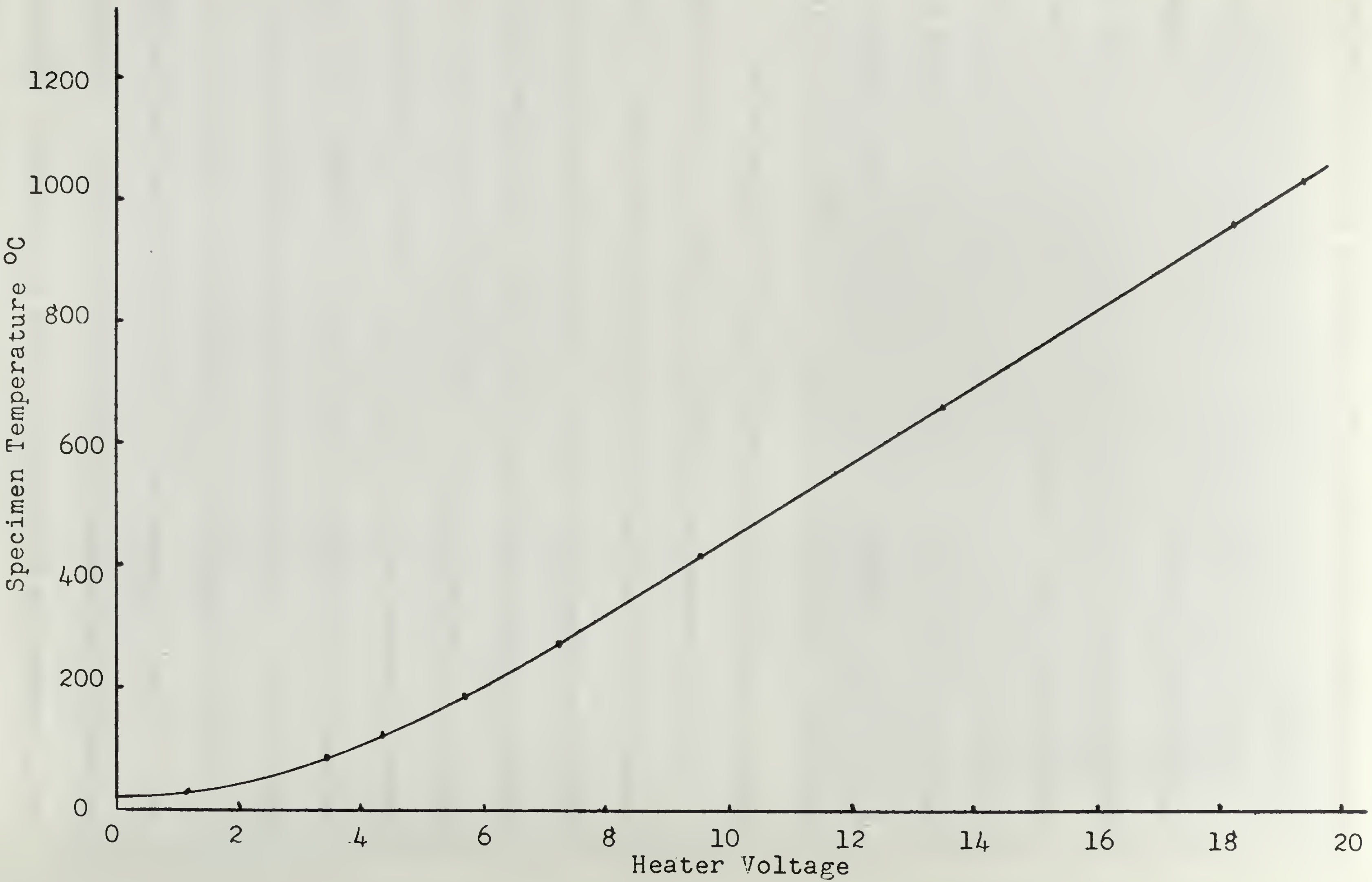


Figure 20. Furnace Calibration Curve

This diamter is accurate enough that interplanar spacings of lines can be measured with a millimeter ruler without introducing an appreciable error. From the interplanar spacings of the most intense lines, it is still possible to use this camera for identification work; although it is better suited for phase transformation and melting point determinations.

8) Summary and Conclusions

The initial objectives of this research were to extend the useful temperature range of the continuous powder photograph camera and to calibrate it so that more precise temperature measurements could be made. By means of the helipot arrangement previously discussed, the temperature of a particular point of interest can be measured with an accuracy of ± 5 degrees centigrade. This is sufficient to determine the starting point for more precise work using a conventional single photograph high-temperature camera.

In order to extend the maximum temperature available beyond the 1045 degree centigrade point, provisions for a controlled atmosphere should be made. This is one area where further study is needed. Another area where further modifications are required is in the film cassette. In order to determine precise lattice parameters, some provision for back reflections must be made. A W-20 film cassette of the type currently used on the Weissenberg camera could be easily installed on this camera by redesigning the film carriage and heater water jacket. Since the Weissenberg film cassette uses a paper light shield, the aluminum alloy absorption problem with the present cassette would be eliminated at the same time that back reflections are gained. The modification to use the Weissenberg cassette would require a minimum of redesigning, but would greatly extend the usefulness of the camera. As an example, internal standards for furnace calibration could be used when compatible with the material to be investigated.

The continuous photograph powder x-ray camera is ready in its current configuration for further research into the phase transformation field of both metals and non-metals. It is particularly adapted for this type research and should be a valuable tool for phase transformation studies.

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APPENDIX

This appendix contains working drawings for a furnace and a helipot support device for a high temperature continuous photograph powder x-ray camera.

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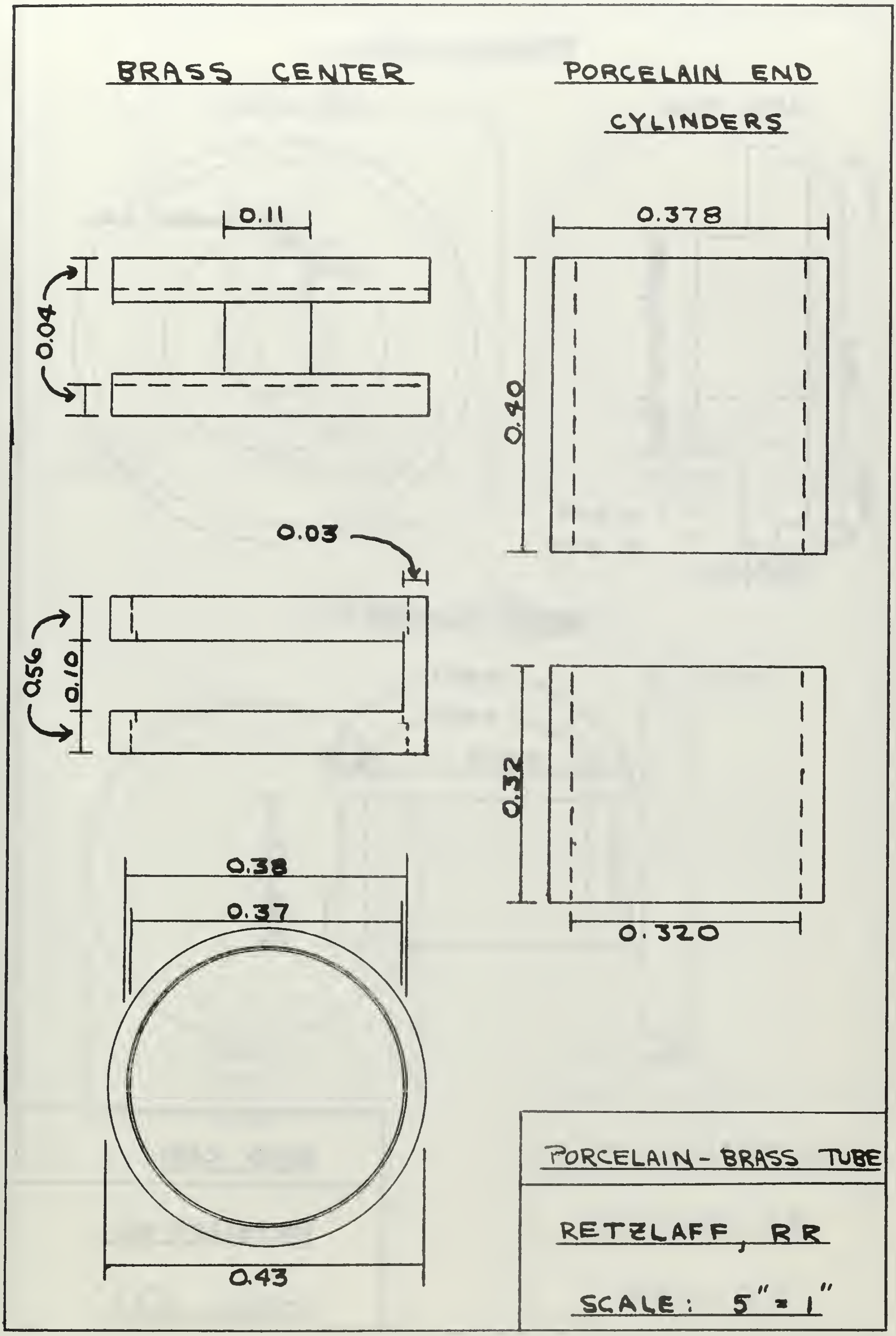
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0.03
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0.32
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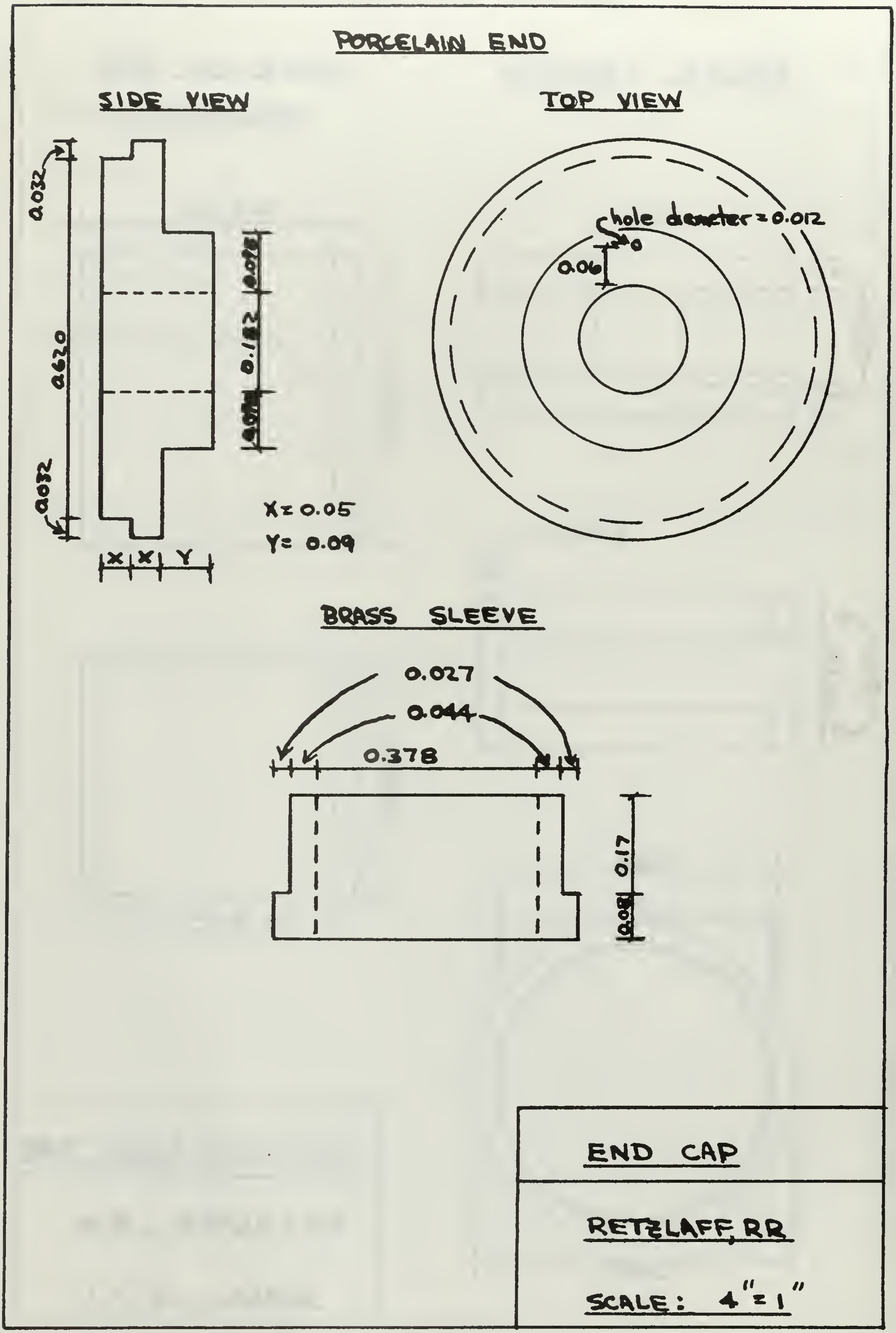
PORCELAIN-BRASS TUBE
RETZLAFF, RR
SCALE: 5" = 1"


PORCELAIN END
SIDE VIEW
TOP VIEW
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0.620
0.032
0.098
0.182
0.098
X X Y
X= 0.05
Y= 0.09
hole diameter = 0.012
0.06
BRASS SLEEVE
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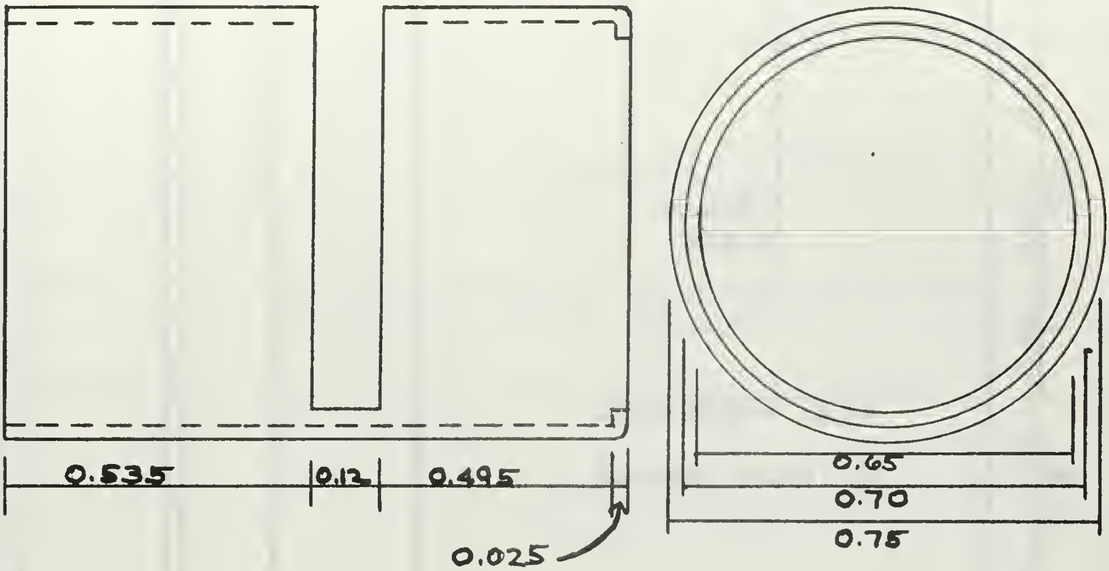
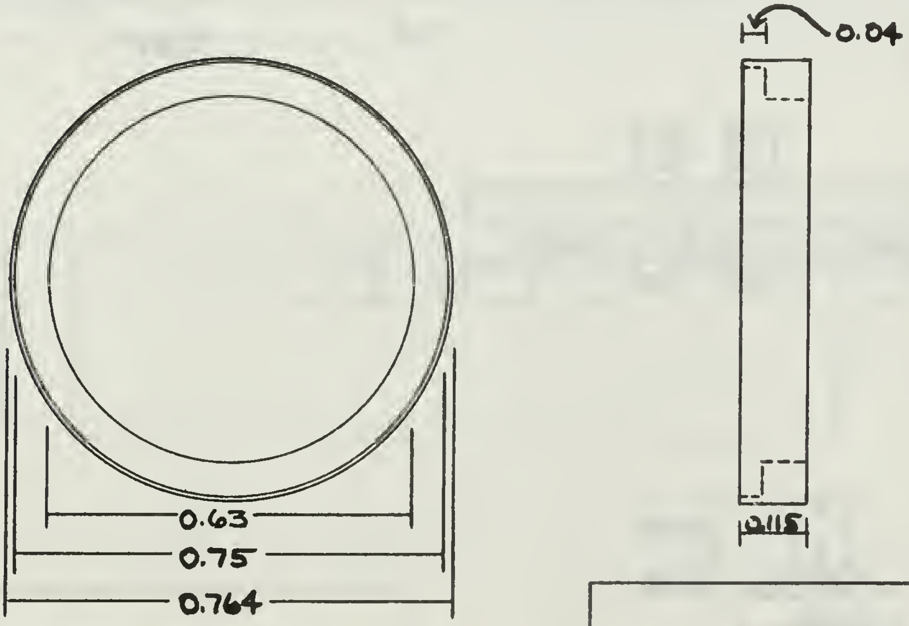
0.044
0.378
0.17
0.08
END CAP
RETZLAFF, RR
SCALE: 4" = 1"

COPPER TUBE



BRASS CAP



COPPER CARTRIDGE

RETZLAFF, RR

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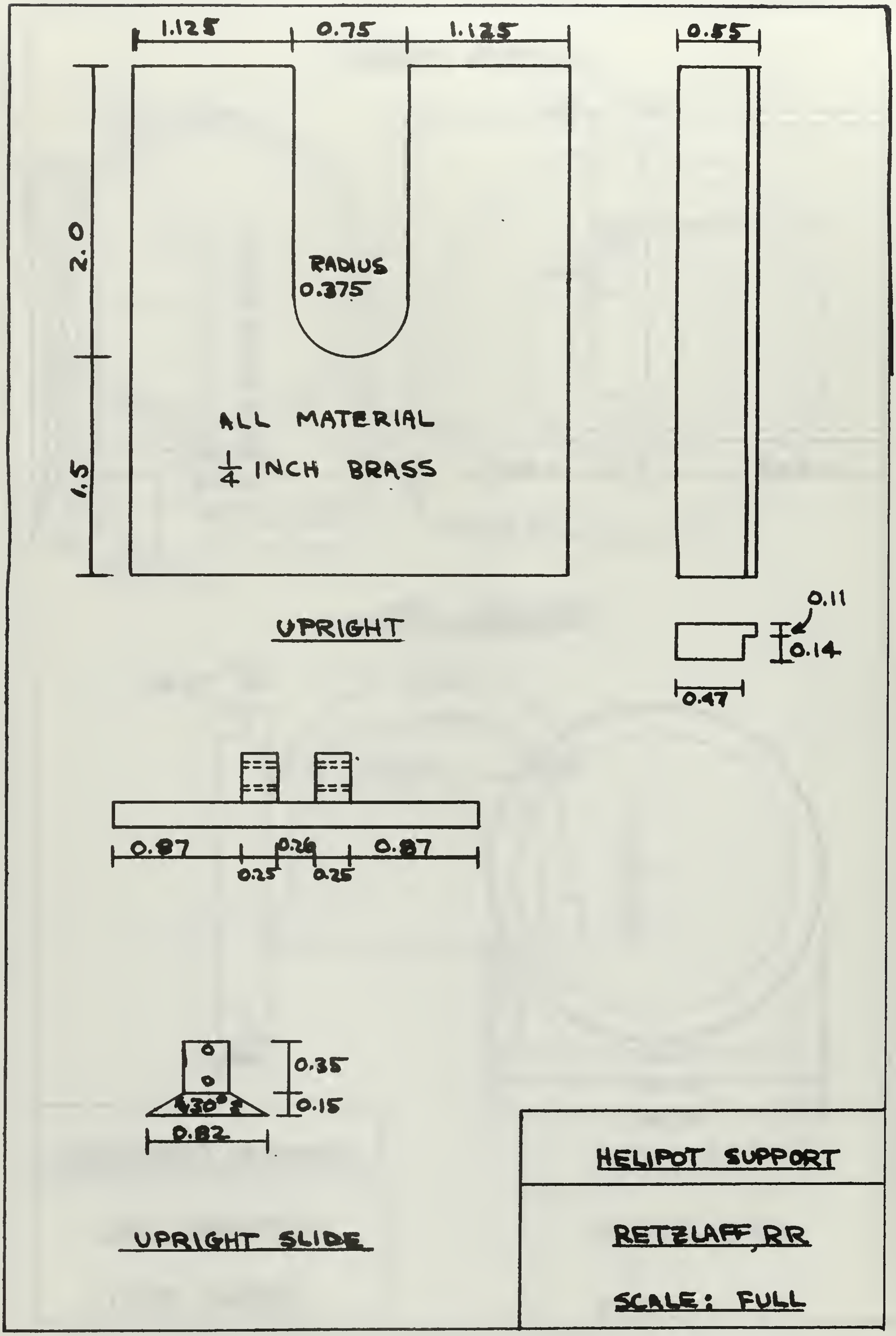

HELIPOT SUPPORT

RETZLAFF, RR

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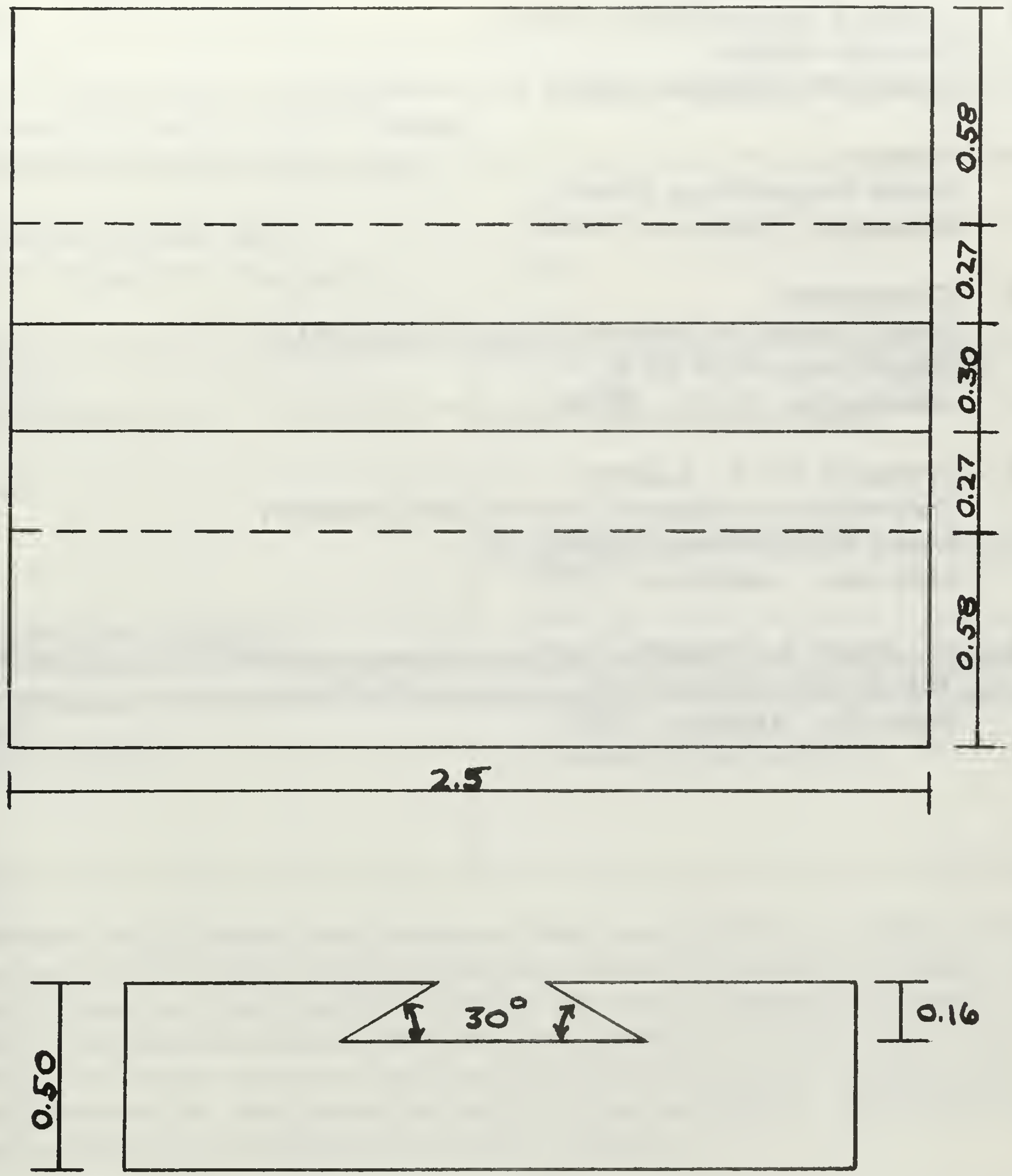

BRASS MATERIAL

HELIPOT SLIDE MOUNT

RETZLAFF, RR

SCALE : 2" = 1"

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DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*

Naval Postgraduate School
Monterey, California 93940

2a. REPORT SECURITY CLASSIFICATION

Unclassified

2b. GROUP

3. REPORT TITLE

FURNACE DESIGN AND CALIBRATION OF A HIGH-TEMPERATURE CONTINUOUS PHOTOGRAPH POWDER X-RAY CAMERA

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

Thesis

5. AUTHOR(S) *(Last name, first name, initial)*

RETZLAFF, Robert R., Lieutenant, U. S. Navy

6. REPORT DATE June 1967	**7a. TOTAL NO. OF PAGES** 66	**7b. NO. OF REFS** 31

8a. CONTRACT OR GRANT NO.

b. PROJECT NO.

c.

d.

9a. ORIGINATOR'S REPORT NUMBER(S)

9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

10. AVAILABILITY/LIMITATION NOTICES

[redacted]

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

13. ABSTRACT

Several modifications are described that were made to an x-ray camera for continuous powder photographs during programmed temperature changes. The original furnace was redesigned to extend the useful temperature range of the camera. Drawings delineating the size and operation of this furnace are included. All of the methods for furnace calibration are summarized, with special emphasis on the transition-melting point technique. Materials used for furnace calibration are tabulated for easy reference.

The camera with the new furnace was calibrated so that more precise high temperature measurements could be made. Further work disclosed that several more desirable features are needed; and these are given in the summary.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
HIGH TEMPERATURE X-RAY CAMERA X-RAY CAMERA FURNACE DESIGN CONTINUOUS PHOTOGRAPH POWDER X-RAY CAMERA FURNACE CAMERA CALIBRATION METHODS						

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